A MODERN ELECTROTHERMAL VAPORISATION INSTRUMENTATION (ETV-ICP-OES) AND IT´S APPLICATION TO ENVIRONMENTAL AND BIOLOGICAL SAMPLES

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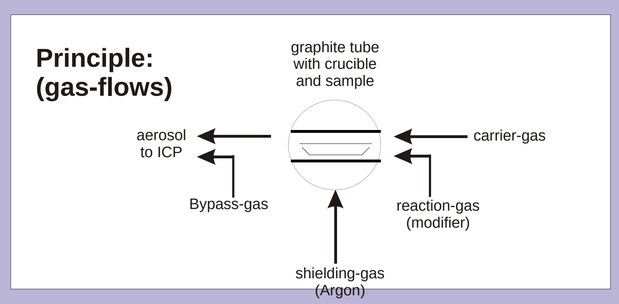
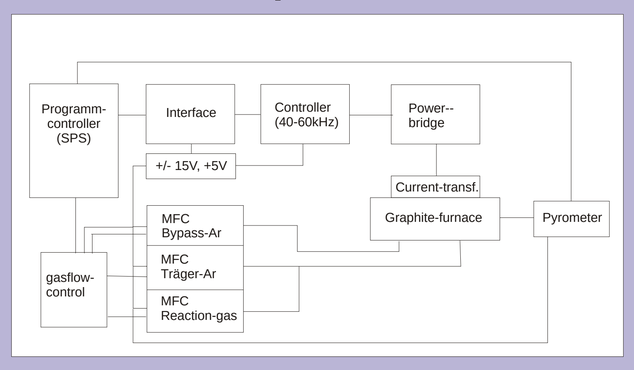
ETV: Principle of operation

Temperature-controlled evaporation in a graphite crucible in a graphite-tube furnace with Argon atmosphere (up to 3000°C),
electronic controlled addition of a reaction-gas (modifier),transport of the aerosol to the ICP-plasma by optimised gas guide
with high transport-efficiency. Integrated microprocessor-control with graphic LCD-display, electronic gas-flow-control-
and mixing, synchronisation by electronic interface. Real-time touchless temparature-measurement of the crucible and
automatic temperature control. Automated sample-handling by autosampler with up to 50 positions, microbalance.

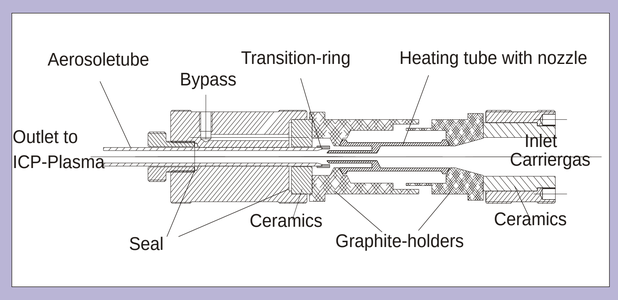


Schematic: computer, controller, powersupply, gas-controls, online-temperature-control



Graphite-furnace, new design

stable up to 3000°C, chemical inert materials, easy handling and maintenance,minimised seals, reduced number of working parts



ETV-analysis: example for a typical procedure

sample preparation: crashing, grinding, drying (if necessary)

weight: 0,5-5 mg

calibration, weight-dependent with 1-2 standards max.6 weights

analysis of the samples 3 weights per sample)

alternative calibration using liquid samples

Simultaneous analysis of all elements, up to 30 spectral lines

transfer of the results to the evaluation-program

assessment of calibration-functions and control-analysis (standards)

documentation of the results

Examples of application

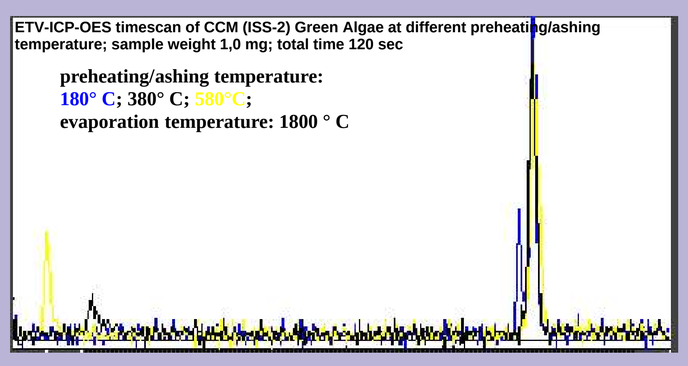
The results of analysed element-concentrations were calculated basing on the shown calibration functions. The points on these common calibration functions (dried liquid standards and solid sample material) show an excellent correlation and thereby prove the correctness and the convincing features of this method. The examples themselfes were selected as best results from a greater number of analyses.

The materials were:

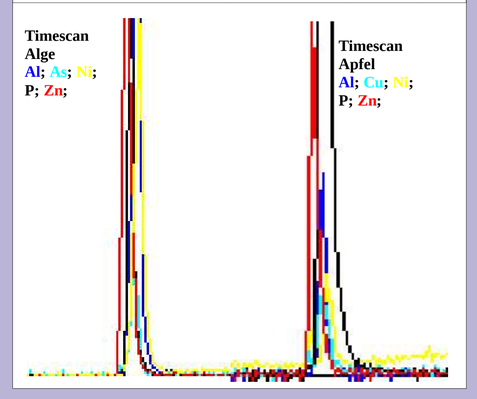
- Pure Graphite (home standards)
- Candidate CRM Green Algae (ISS-2)
- NIST SRM 1515 Apple Leaves

Concentrations:

Element	Candidate RM Green Algae	NIST CRM 1515 Apple Leaves
Al		286 9 µg/g
As	4,5+/- 0,5 µg/g	0,038+/- 0,007 µg/g
Cu		5,64+/- 0,24 µg/g
Ni	33+/- 6 µg/g	0,91+/- 0,12 µg/g
P		0,159+/- 0,011 %
Zn	35,2+/- 3,4 µg/g	12,5 0,3 µg/g



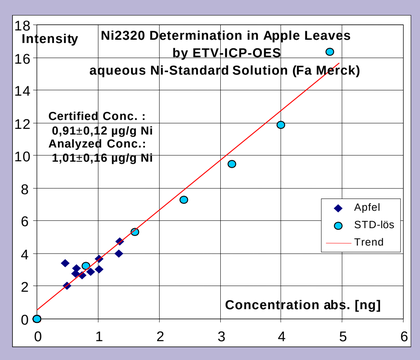
ETV-ICP-OES timescans NIST SRM 1515 Apple Leaves and CCM (ISS-2) Green Algae; sample weight 1,0 mg

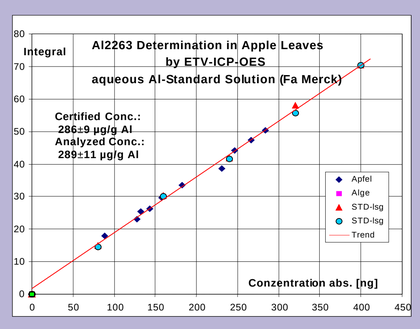


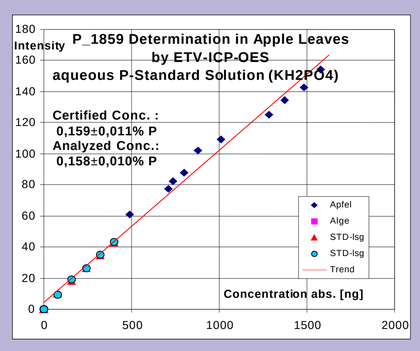
Tab. Experimental setup for ETV-ICP-OES:

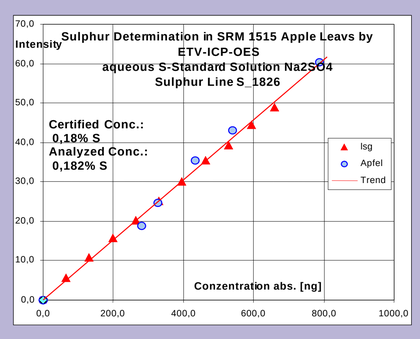
ICP-Spectrometer	IRIS-AP Thermo Jarrell Ash Echelle-polychromator, Argon rinsed Echelle grating 60 grooves/mm
Spectral data	Focal distance: 381mm Resolution: at 200 nm width of one pixle 0,0035 nm Spektral range: 175-1050nm
Signal detection	CID-camera Active surface of CID chip:14,3×14,3mm (512 x 512 pixle)
ICP	axially plasma; RF generator: 1150 W at 27,12 Mhz
ETV-unit	ETV 4000 P.Perzl; Spectral Systems; Power supply: max power 400 A; end-on stream system; Furnace control: inside temperature controled

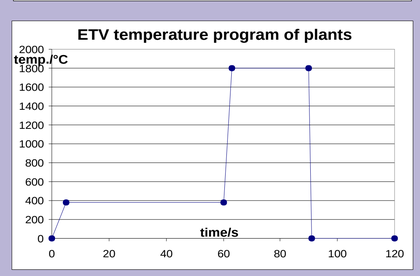
Al, Ni, P, S in Apple Leaves



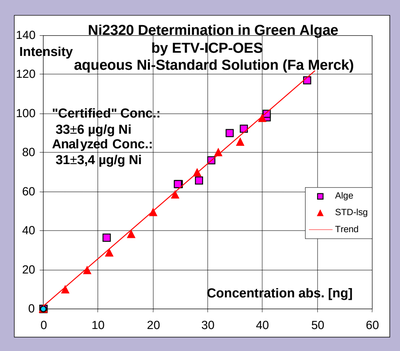


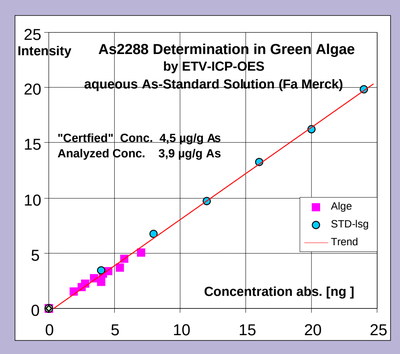


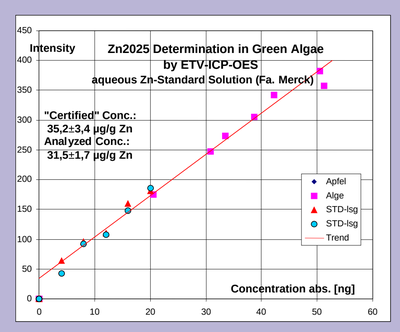




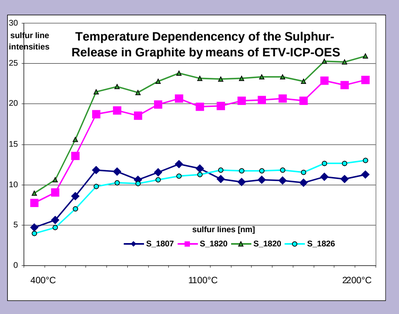
As, Ni, Zn in Green Algae

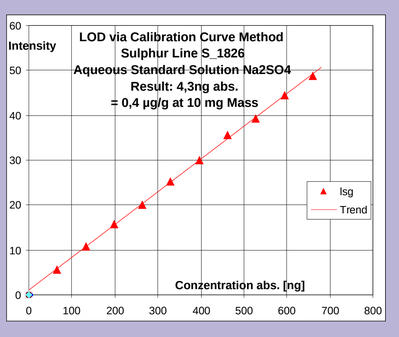


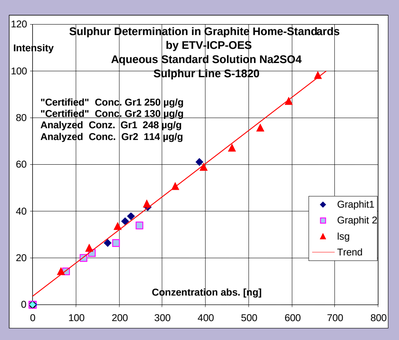




S in Graphite







Conclusion:

Modern ETV-ICP configurations
are an excellent and cost-effective tool for easy, fast
and precise direct solid sample multi-element analysis in
a wide area of applications. The field of applications ranges from anorganic materials like ceramics and geological samples up to environmental samples and biological materials like the shown examples.
The presented results demonstrate the easy and uncomplicated possibilities of calibration via liquid standard solutions compared with the certified plant materials.
The found limits of detection were less than 1ng abs. and the typical reproducibilities better than 5% rel.
A professional ETV-equipment is further more
rationalised by an autosampler with up to 50
crucibles and integrated micro-balance.